

(54) Title
DIRECTLY COMPRESSIBLE PULVERULENT COMPOSITION AND A PROCESS FOR OBTAINING THE SAME

(51)⁵ International Patent Classification(s)
A23L 001/09 A23G 003/30

(21) Application No. : 89760/91

(22) Application Date : 13.12.91

(30) Priority Data

(31) Number (32) Date (33) Country
90 15708. 14.12.90 FR FRANCE

(43) Publication Date : 18.06.92

(71) Applicant(s)
ROUQUETTE FRERES

(72) Inventor(s)
MICHEL SERPELLONI; ALAIN CROISIER

(74) Attorney or Agent
WATERMARK PATENT & TRADEMARK ATTORNEYS, Locked Bag 5, HAWTHORN VIC 3122

(57) Claim

1. Directly compressible pulverulent composition based on xylitol, characterised in that it has a compressibility, determined in a test A, above 70 N, preferably above 80 N.

3. Composition according to Claim 1 or Claim 2, characterised in that it contains at least one additive selected from saccharides, oligosaccharides and polysaccharides and their corresponding hydrogenated compounds.

6. Process for the fabrication of the directly compressible pulverulent composition according to any one of Claims 1 to 5, characterised in that a starting material substantially consisting of xylitol, that is to say present in a quantity greater than or equal to 60%, preferably to 80%, and at least one additive, is subjected to an extrusion treatment comprising a thermal treatment zone and at least one extrusion die, the rate of feed of starting material into the apparatus and the parameters of the extrusion treatment, namely the temperature inside the thermal treatment zone, the diameter of the extrusion die and the speed of transport of the starting material inside the thermal treatment zone, being so chosen that the xylitol/additive mixture will be partly molten at the outlet of the die and before its discharge from the latter.

(11) 89760/91

-2-

9. Use of the pulverulent composition according to any one of claims 1 to 5 as sweetening filler in tablets or in articles of confectionery, in particular of the chewing gum type.

AUSTRALIA

Patents Act 1990

ORIGINAL
COMPLETE SPECIFICATION
STANDARD PATENT

Application Number:

Logged:

Invention Title:

DIRECTLY COMPRESSIBLE POLYMER CELL COMPOSITION AND A
PROCESS FOR OBTAINING THE SAME

The following statement is a full description of this invention, including the
best method of performing it known to :- us

DIRECTLY COMPRESSIBLE PULVERULENT COMPOSITION AND A
PROCESS FOR OBTAINING THE SAME

The present invention relates, by way of novel industrial product, to a directly compressible pulverulent composition based on xylitol.

It also has as its object a process for the preparation of this pulverulent composition.

Further, it relates to the use of this pulverulent composition as sweetening filler in tablets and articles of confectionery of the chewing gum type.

Xylitol, which is a pentavalent alcohol sugar is a sweet crystalline product, white in colour, odourless and soluble in water. In crystalline form, it has a negative dissolving heat and thereby produces an agreeable refreshing or cooling effect in the mouth. This property is particularly advantageous in pharmaceutical or food tablets.

In addition to its cooling effect, xylitol has interesting sweetening qualities. If one takes sucrose as reference point and attributes to it a sweetening value of 1, xylitol is found to have a sweetening value of the same order whereas for more traditional polyols such as sorbitol or mannitol the values are, respectively, 0.60 and 0.45.

Like other alcohol sugars already used in the food industry and pharmacy, xylitol is an advantageous sucrose substitute for diabetics.

Moreover, xylitol has an interesting property for dental health, in which it differs from other known polyols. It is in fact not only acariogenic, that is to say it can not serve as substrate for bacteria present in the mouth cavity, but it also plays a role in preventing dental caries.

It is thus on several grounds that the use of xylitol would be justified as binder and diluent in the fabrication of tablets.

10 It is well known that certain powders are by their nature difficult to compress. This is unfortunately the case with crystallised pulverulent xylitol, which therefore cannot be obtained as sufficiently hard tablets. Its limit of compressibility is relatively low, so that it favours the appearance of the phenomenon of capping, that is to say splitting of the tablet in a diametric plane which eliminates all its cohesion. Such a tablet is very friable and has the tendency to break into two or more parts when tested for hardness or even when ejected from the press.

20 One of the known techniques for conferring the capacity for compression on powders is moist granulation. This is a expensive and relatively complicated process to carry out in which supplementary additives are used as binders, such as starch, cellulose or gelatine. As regards xylitol, it is well known that this product is very difficult to granulate under moist aqueous conditions due to its hygroscopic character and its very high solubility in water. It is therefore necessary to use non-aqueous solvents such as polyethylene glycol or ethanol, which, however, do not have the same advantages as water from the aspect of economics and toxicity. Moreover, the

compressibility of xylitol granules prepared by this method is not good, see Acta Pharmaceutica, Fennica 87, pages 61 to 73 (1978) and 91, page 48 (1982).

5 The technique of so called direct compression without previous treatment of the powder thus remains the most advantageous method for the pharmaceutical industry and food industry, in particular from an economical point of view.

10 In attempting to render xylitol directly compressible, it was initially proposed to mix crystallised pulverulent xylitol with different known binders such as microcrystalline cellulose, see Acta Pharmaceutica, Fennica 91, pages 47-54 (1982). Apart from the fact that this compound is insoluble, so that it would be difficult to use for
15 tablets which are to be sucked, which are the most common form of tablets used in the food industry, it should be noted that microcrystalline cellulose is present in a proportion of 10 to 50% by weight, based on the xylitol. It therefore has the tendency to mask the expression of certain advanta-
20 geous properties of crystalline xylitol such as its sweetening power, its cooling effect and its agreeable taste. This obviously reduces the interest in using compressed crystallised xylitol. Moreover, the economical incidence of incorporating microcrystalline cellulose is not to be neglected
25 as this is a relatively expensive additive. Lastly, the presence of binder is not greatly favoured by the manufacturers of compressed products, in particular for pharmaceutical purposes.

30 It has also been proposed in French Patent Application No.2 336 123 to produce tablets which are to be chewed from a dry mixture of xylitol in a quantity of 10 to 80% by weight and a polyol in a quantity of 10 to 80% by

weight, based on the weight of the tablet. The polyol may be sorbitol, mannitol or a mixture of these two. It appears from the description of the said Patent Application that the polyol amounts to at least 50% by weight of the xylitol. The latter therefore cannot be regarded as the main constituent of the powder to be compressed. Under these conditions, it is not possible to obtain optimum benefit from all the advantages of xylitol.

European Patent Application No. 305 356 describes a process for the preparation of granulated products suitable for direct compression. In this process, a pulverulent product, which may be xylitol, is brought into contact under conditions of stirring with a liquid consisting of the same product in the molten state, and the mixture obtained is then rapidly cooled.

Due to this last arrangement, the product obtained is present to a non-negligible proportion in an amorphous form. This may entail certain disadvantages, in particular a loss of cooling effect, a greater hygroscopicity and hence a tendency of the powder to clump together, and low compressibility. It will also be noted that this process is relatively delicate to carry out. Moreover, it has been verified that the granular product obtained has poor characteristics of friability and cannot be used for preparing tablets having properties of compression, in particular hardness, compared with tablets obtained with conventional crystallised xylitol.

In a process for agglomerating very fine crystallised xylitol powder by means of a sorbitol syrup under vigorous stirring, the owner of European Patent Application No. 329 977 proposes a binding agent and diluent which can be used

for direct compression, the granules having a particle size of about 0.1 to 1 mm and containing 95 to 98% by weight of xylitol, 1 to 5% by weight of sorbitol, 0 to 2% by weight of other polyols and less than 1% by weight of water. This agent has an apparent density ("bulk density") of from 0.7 to 0.8 g/cm³.

Previous crushing of xylitol for obtaining a fine particle size, agglomeration with a sorbitol syrup having a low solids content and final drying of the powder do not amount to a process having the desired simplicity and they constitute additional operations which increase the operating costs.

Besides, it has been found that the results obtained in terms of hardness of the tablets prepared with this binding agent and diluent are not satisfactory.

It is on the basis of these last findings and in view of all the disadvantages of the other processes known in the art that the Applicant has sought to provide a pulverulent composition for direct compression based on xylitol, that is to say a composition mainly consisting of this polyol, and having improved properties of compressibility and flow compared with existing pulverulent products containing xylitol.

As a result of numerous studies and tests, the Applicant has succeeded in preparing a directly compressible pulverulent composition based on xylitol, characterised by a compressibility, determined by a test A, above 70 N and preferably above 80 N.

This value for compressibility is surprisingly and unexpectedly greatly superior to the values obtained with

xylitol-based compositions hitherto known until now.

The proportion of xylitol in the composition is preferably greater than or equal to 60% by weight and more preferably 80% by weight.

5 Test A consists of measuring the force, expressed in
Newtons, representing the compressibility of the composi-
tion under investigation which is necessary for crushing
a tablet prepared from the said composition, that is to
say f o r p r o d u c i n g t h e a p p e a r a n c e o f
10 fracture lines inside the mass of the tablet, this force
thus corresponding to the resistance to crushing of the
tablet which is cylindrical in form with flat surfaces
having a diameter of 13 mm and with a thickness of 4 mm
and a weight of 0.717 g, that is to say an apparent
15 volumetric mass or density of 1.35 g/ml, the said force
being exerted against the peripheral surface of the tablet
in the direction of the axis of revolution of the tablet
by means of a movable s t o p applying a thrust against
the said surface along a generatrix. During this test, the
20 tablet is held against a fixed s t o p also applied
against the peripheral surface of the tablet along a
generatrix which is diametrically opposite to that against
which the movable s t o p is applied.

These tablets are prepared by adding 2% by weight of
25 lubricant, namely magnesium stearate, to the composition
under investigation.

These two products are homogenised with one another by
means of a TURBULA T2C mixer (marketed by WILLY A.
BACHOFEN AG, Switzerland) for five minutes at a driving speed
30 of 42 revolutions/minute.

To compress the obtained mixture, a FROGERAIS reciprocating press of type AM is used. This press is equipped with round punches with plane surfaces having a diameter of 13 millimetres.

5 To obtain the characteristics of the tablets mentioned above, the press is adjusted to regulate the amount by which the upper punches press in and the filling volume of the matrix, the latter arrangement being used to fix the desired quantity by weight of the pulverulent mixture,
10 which in the present case is 0.717 g.

The resistance of these tablets to crushing is determined by means of a SCHLEUNIGER 2E durometer (marketed in France by FROGERAIS Establishments).

The composition according to the invention contains at
15 least one additive selected from saccharides, oligosaccharides and polysaccharides and their corresponding hydrogenated compounds, that is to say organic molecules characterised by the presence of carbon chains carrying hydroxyl groups and aldehyde, ketone or acid functions.

20 The additives chosen are preferably sugars such as aldoses or ketoses and their hydrogenated derivatives, for example sorbitol, mannitol or maltitol and polysaccharides, in particular polymers of glucose such as maltodextrins having a Dextrose Equivalent below 20, or glucose syrups,
25 and their hydrogenated derivatives.

According to one advantageous arrangement of the invention, the additive is selected from the following list of compounds, which is not limiting: Sorbitol, maltitol, mannitol, maltodextrin. In a preferred embodiment for carrying
30 out the invention, the additive selected is sorbitol.

It goes without saying that the invention is not limited to the use of only one additive in addition to xylitol but also covers any composition containing several additives differing in nature.

- 5 The present invention also relates to a process for the preparation of the said pulverulent composition based on xylitol.

According to this process, a starting material substantially consisting of xylitol, that is to say containing a
10 quantity of xylitol greater than or equal to 60%, preferably 80%, and at least one additive, is subjected to extrusion treatment inside an extrusion apparatus containing a heat treatment zone and at least one extrusion die, the rate of supply of xylitol and additive to the apparatus and the
15 parameters for the extrusion treatment, namely the temperature inside the heat treatment zone, the diameter of the extrusion die and the speed of transport of the starting material inside the heat treatment zone, being selected so that the mixture of xylitol and additive will
20 be partially melted at the outlet of the die and before it leaves the die.

The pulverulent composition based on xylitol according to the invention is also defined as being a pulverulent composition obtainable by the process according to the
25 invention.

The above said installation is preferably of the double screw type having at least one extrusion die and the parameters for the extrusion treatment are selected so that the starting material is at a temperature of from 75 to

110°C inside the die and before its discharge from the die, the said temperature depending on the nature and quantity of the additive or additives put into the process.

5 This temperature may easily be determined by the man of the art as he knows that the melting temperature of xylitol is in the region of 92°C and the proportion of molten starting material is preferably from 30 to 90%, more preferably from 50 to 80%, so that the
10 product obtained at the extrusion outlet has a viscosity which enables it to be easily and rapidly subjected to the subsequent operations of the process.

15 If the pulverulent composition contains a single additive and the latter is sorbitol, the temperature of the starting material inside the die and before the outlet of the die is preferably from 80 to 105°C.

If the single additive is maltitol, the said temperature is preferably from 80 to 100°C.

20 Advantageously, the xylitol and additive which constitute the starting material to be extruded are in a pulverulent form and may or may not be crystallised.

The invention further relates to other arrangements preferably used at the same time which will be more
25 particularly described below, and it will be better understood with the aid of the description given below with the attached Figures 1 and 2 and examples.

Figure 1 is schematic section through an extrusion apparatus suitable for use in the process according to the

invention.

The following method or an equivalent method is employed for fabricating a directly compressible pulverulent composition based on xylitol according to the invention:

- 5 The starting material which is subjected to the extrusion treatment by the process according to the invention consists, for example, of a mixture of 90% by weight of xylitol powder and 10% by weight of sorbitol powder.

10 In practice, crystallised xylitol and sorbitol obtained by the techniques conventionally employed in this field are used.

The extrusion apparatus advantageously consists double screw-type extruder comprising, as shown in Figure 1:

- a feed device, in particular a feed hopper 1,
- 15 - a mixing device M comprising an endless double screw system 2 inside a housing 3 in particular of nitrided steel rotated by a not shown mechanism,
- an outlet comprising one or more extrusion dies 4 of differing forms,
- 20 - means for thermal regulation 5 to control the temperature of the mixing zone, the said means 5 consisting on the one hand of heating means, for example, formed by electric resistors or by a heating system operating by induction or steam, and
- 25 - on the other hand of not shown cooling means arranged outside the housing or on the inside and consisting, for example, of cooling coils located in the housing, and/or of a circulation of cooling fluid inside the screw.

30 The starting material introduced into the mixing zone

from the feed system is subjected to shearing forces and intense mechanical friction by the compression in the turns of the screw and at the same time to heating induced by the heating means employed.

- 5 The extrusion consequently consists of a thermomechanical treatment.

It should be noted that good results have been obtained with a double screw-type extruder marketed under the name of "BC 82" by CENTRAL Company. The two screws intermesh and turn in
10 the same sense. The mixing zone is heated by induction or cooled by circulation of a cooling fluid in a coil so that the temperature can easily be regulated.

The main advantage of this heating method is the flexibility in use and the ease of control by means of a simple
15 control loop (thermocouple/control device of the heating means by induction of the cooling means).

The installation used in this example comprises four cylindrical extrusion dies 5 mm in diameter.

The temperature of the heat treatment zone is obtained by
20 imposing a predetermined value on the control system. In the case of the extrusion apparatus in question, this value is from 75 to 110°C, preferably from 80 to 105°C.

The mechanical characteristics of the screws and their speed of rotation are chosen so that the residence time of
25 the starting material inside the heat treatment zone will be from 5 to 300 s.

As a result of the choice of all these parameters, the temperature of the starting material which has been

subjected to the treatment is from 75 to 110°C inside the dies and before exit from the latter.

The coextruded mixture based on xylitol obtained at the outlet from the extrusion apparatus is then subjected
5 successively to:

- cooling,
- crushing and
- sifting.

According to another feature of the present invention, the
10 pulverulent composition according to the invention is found to be particularly suitable as sweetener for confectionery and in particular confectionery of the chewing gum type.

One of the classical problems confronting manufacturers
15 in the industrial production of chewing gums is the lack of "machinability" of the mass of basic gum and the processed sweetening filler. This mass is conventionally handled at a temperature of 40 - 50°C. It is important that the mass should be sufficiently soft at this temperature to enable
20 all the components of the chewing gum (basic gum, sweetening filler, flavouring substances, colouring substances...) to be mixed homogeneously but not too soft in order to avoid problems of sticking during the stages of mixing, forming and cutting.

25 The sweetening filler has a significant influence on the viscosity of the processed work mass. It is known, for example, that if the sweetening filler is a powder, good hardness at a high temperature can be obtained if the granulometry is very fine. The disadvantage of these fine powders is their
30 very high hygroscopicity, with the result that they tend to clump together during storage.

It is surprisingly and unexpectedly found that the composition according to the invention enables chewing gums to be obtained which have very good hardness both in the hot and cold state without requiring very fine granulometries. Moreover, the organoleptic quality of these chewing gums is entirely satisfactory.

The present invention thus also relates to the use of the pulverulent composition based on xylitol according to the invention as sweetening filler in confectionery of the chewing gum type as well as relating to the confectionery products thus obtained.

The very sweet flavour, the cooling effect and the cario-static effect of xylitol are properties which are particularly appreciated for the use of this product in confectionery of the chewing gum type.

The examples given below illustrate the significant differences between the pulverulent compositions according to the invention and products based on xylitol for direct compression either produced according to the prior art or produced for comparison purposes by the Applicant Company, and the advantages obtained by the use of the said pulverulent compositions in confectionery of the chewing gum type.

EXAMPLES RELATING TO THE USE UNDER COMPRESSION

The following are used in these examples:

- Five different samples of the pulverulent composition according to the invention, indicated by the references a, b, c, d and e;
- a sample of XYLITOL DC marketed by CULTOR Company and indicated by the reference f;

- a sample of extruded xylitol powder having a minimum chemical purity of 99% by weight of dry substance and indicated by the reference g;
- a sample of pulverulent xylitol marketed by ROQUETTE Company and having a minimum chemical purity of 99% by weight of dry substance, indicated by the reference h;
- three samples of pulverulent mixtures containing xylitol powder and sorbitol powder marketed by ROQUETTE Company, having a chemical purity of, respectively, 99% and 96% by weight of dry substance, in which the xylitol powder and sorbitol powder are mixed together in the dry state; the proportions by weight of these xylitol/sorbitol mixtures are, respectively, 50/50 for the sample indicated by the reference i, 90/10 for the sample indicated by the reference k and 95/5 for the sample indicated by the reference l ;
- a sample j of compressible xylitol prepared by the process described in Patent Application EP.0 305 356 by mixing 300 g of molten xylitol at a temperature of about 100°C and 700 g of crystallised xylitol;
- and two samples of pulverulent mixtures consisting of xylitol and sorbitol which have been extruded separately and then mixed together in the dry state; the minimum chemical purities of the xylitol and sorbitol are, respectively, 99% and 96% by weight of dry substance; the proportions by weight of these xylitol/sorbitol mixtures are, respectively, 90/10 for the sample indicated by the reference m and 98/2 for the sample indicated by the reference n.

The five samples f to j belong to the prior art and the

four samples k to n were selected and prepared by the Applicant for comparison purposes.

1. Preparation of five samples a-e according to the invention

5 For the three samples a-c, the additive chosen is sorbitol of the type marketed by the Applicant under the trade mark NEOSORB(R) P60. The quantities of sorbitol are, respectively, 10%, 5% and 2% by weight based on the composition for samples a, b and c. For sample d, the additive chosen
10 is pulverulent maltitol present in a proportion of 10% by weight, based on the composition.

15 Lastly, for sample e, the additive chosen, which is present in a proportion of 10% by weight, based on the composition, consists of a maltodextrin obtained by enzymatic hydrolysis of starch and having a Dextrose Equivalent or D.E. (number of grams of reducing sugars expressed as dextrose to 100 grams of dry product) of 12, marketed by the Applicant under the Registered Trade Mark GLUCIDEX(R) 12.

20 The xylitol used in each of the samples a to e is obtained by crystallisation in water and contains a minimum of 99% of xylitol by weight, based on the solids content.

25 Samples a to e are prepared by introducing a homogeneous pulverulent mixture of xylitol and additive into the extruder in the proportions indicated above. The extruder used is of the type mentioned above, that is to say "BC 82" of CLEXTRAL Company.

The speed of the screws is controlled so that the output of the installation is 200 kg/hour and the time taken for the starting material to pass through the apparatus is

about 30 s.

The predetermined temperature of the heating system is programmed to a value from 85 to 110°C, varying according to the nature and quantity of the additive.

5 For samples a to e, the temperatures chosen are as follows:

- sample a \approx 90°C,

- sample b \approx 92°C,

- sample c \approx 93°C,

10 - sample d \approx 90°C,

- sample e \approx 92°C.

At the exit from the extruder and after cooling, the coextruded mixtures of xylitol/additives are in the form of small sticks which are crushed in a hammer mill.

15 The fraction held back has a particle size above 50 microns, more generally above 100 microns.

2. Tests

For each of the samples a to n tested, an average particle size of from 400 to 900 microns was selected.

20 Using the CARR method as described by R.L.CARR in Chem. Eng. 72, No.163, 168 (1985) and Chem. Eng. 72, No.2, 69-73 (1985), the flow index and the density of powders a to n are measured. The apparatus used for this test is the one known under the trade mark HOSOKAWA POWDER TESTER
25 manufactured by MICROMERITICS, Osaka (Japan).

In addition, the friability of some of the pulverulent compositions a to m are determined. This property is

characterised by the percentag of particles which have not resisted crushing in a friability measuring apparatus. In the present case, the apparatus of Trade Mark ERWEKA TA was used. This apparatus contains five identical steel 5 balls 1.7 cm in diameter, each weighing 18.87 g. 15 g of a fraction measuring 400 to 500 microns of the tested powder are introduced and the apparatus is rotated at the rate of 25 revolutions per minute for 15 minutes. At the end of the crushing operation, the proportion held back by a 10 sifter having a mesh of 351 microns, expressed as a percentage, is determined by weighing. The friability then corresponds to the amount required to make this value up to 100 g. The larger the number so obtained, the greater is the friability.

15 The compressibility of these samples is then determined by test A defined above.

The results of these measurements of compressibility by test A and the mean granulometry, the flow indices and the friability of the tested samples are summarized in the 20 Table below.